FORM PTO-1390
TRADEMARK OFFICE
(REV 9-2001)

U S DEPARTMENT OF COMMERCE PATENT AND

TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. 371

ATTORNEY'S DOCKET NUMBER	
TS0854 US	
U S APPLICATION NO (If known, see 37 CFR 1 5)	
10/031753	

INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP00/07174	25 July 2000	26 July 1999

TITLE OF INVENTION:
BREAKING OF OIL/WATER EMULSION

APPLICANT(S) FOR DO/EO/US:
VAN DEN BERG, Franciscus, G.A.; JANSEN, Arie; and STAMPS, Paulus, A.

Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☐ This is an express request to begin national examination procedures (35 U.S.C. 371(f)). The submission must include items (5), (6), (9) and (21) indicated below.
4. ☐ The US has been elected by the expiration of 19 months from the priority date (PCT Article 31).
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2)).
   a. ☐ is attached hereto (required only if not communicated by the International Bureau).
   b. ☒ has been communicated by the International Bureau.
   c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US).
6. ☐ An English language translation of the International Application as filed (35 U.S.C. 371(c)(2)).
   A ☐ is attached hereto
   b. ☐ has been previously submitted under 35 U.S.C. 154(d)(4).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)).
   a. ☐ are attached hereto (required only if not communicated by the International Bureau).
   b. ☐ have been communicated by the International Bureau.
   c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
   d. ☒ have not been made and will not be made.
8. ☐ An English language translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☒ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)).
10. ☐ An English language translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11 to 16 below concern document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☒ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☒ A FIRST preliminary amendment.
14. ☐ A SECOND or SUBSEQUENT preliminary amendment.
15. ☐ A substitute specification.
16. ☐ A change of power of attorney and/or address letter.
17. ☐ A computer-readable form of the sequence listing in accordance with PCT Rule 13ter.2 and 35 u.S.C. 1.821-1.825
18. ☐ A second copy of the published international application under 35 U.S.C. 154(d)(4).
19. ☐ A second copy of the English language translation of the international application under 35 U.S.C. 154(d)(4).
20. ☒ Other items or information: postcard for date stamping only

U S APPLICATION NO (if known, see 37 CFR 1.5) 10/031753	INTERNATIONAL APPLICATION NO PCT/EP00/07174	ATTORNEY'S DOCKET NUMBER TS0854 US

21. ☒ The following fees are submitted:

	CALCULATIONS PTO USE ONLY	
Basic National Fee (37 CFR 1.492(a)(1)-(5)):		
Neither international preliminary examination fee (37 CFR 1.482) nor international search fee (37 CFR 1.445(a)(2)) paid to USPTO and International Search Report not prepared by the EPO or JPO **$104.00**		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but International Search Report prepared by the EPO or JPO **$890.00**		
International preliminary examination fee (37 CFR 1.482) not paid to USPTO but international search fee (37 CFR 1.445(a)(2)) paid to USPTO **$740.00**		
International preliminary examination fee (37 CFR 1.482) paid to USPTO but all claims did not satisfy provisions of PCT Article 33(1)-(4) **$710.00**		
International preliminary examination fee paid to USPTO (37 CFR 1.482) and all claims satisfied provisions of PCT Article 33(1)-(4) **$100.00**		
ENTER APPROPRIATE BASIC FEE AMOUNT =	$890.00	
Surcharge of $130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(e)).	$	

CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	1~~0~~ - 20 =	0	x $ 18.00	$	
Independent Claims	1 - 3 =	0	x $ 84.00	$	
Multiple dependent claim(s) (if applicable)			+ $280.00	$	
TOTAL OF ABOVE CALCULATIONS =				$	
☐ Applicant claims small entity status. See 37 CFR 1.27. The fees indicated above are reduced by 1/2. +				$	
SUBTOTAL =				$890.00	
Processing fee of **$130.00** for furnishing the English translation later than ☐ 20 ☐ 30 months from the earliest claimed priority date (37 CFR 1.492(f)). +				$	
TOTAL NATIONAL FEE =				$	
Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). $40.00 per property) +				$ 40.00	
TOTAL FEES ENCLOSED =				$	
				Amount to be refunded:	
				charged:	$930.00

a. ☐ A check in the amount of $_____ cover the above fees is enclosed.

b. ☒ Please charge my Deposit Account No. 19-1800 in the amount of $ 930.00 to cover the above fees. A duplicate copy of this sheet is enclosed.

c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 19-1800. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

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THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re PCT International Application of)
)
VAN DEN BERG, Franciscus G.A., et al)
)
Int. Appl No.: PCT/EP00/07174)
)
Int. Filing Date: 25 July 2000)
)
BREAKING OF OIL/WATER EMULSION) 21 January 2002
)

ASSISTANT COMMISSIONER FOR PATENTS
Washington, DC 20231

PRELIMINARY AMENDMENT UNDER 37 CFR 1.115

Please enter the Article 34 Amendment before entering of the Preliminary Amendment.

IN THE CLAIMS

Amend claims: 2 -10.

2. (Once Amended) The process of claim 1, wherein in step (a) the temperature is raised to a value of between 140-200°C.

3. (Once Amended) The process of claim 2, wherein in step (a) the temperature is raised to a value of between 160-200°C.

4. (Once Amended) The process of claim 3, wherein the temperature of the resulting mixture is raised from a value of between 120-150°C to a value of between 160-180°C by making use of the indirect heat exchange means.

5. (Once Amended) The process of claim 1, wherein the pressure in step (b) is sufficiently high in order to obtain both phases in the liquid state.

6. (Once Amended) The process of claim 5, wherein in step (b) the liquid water phase has a pH of below 7.

7. (Once Amended) The process of claim 6, wherein the pH of the liquid water phase is between 4 and 6.

8. (Once Amended) The process of claim 1, wherein the starting emulsion has a water content of between 1-40% by weight, a surfactants content of between 0.01-5% by weight and an oil content of between 60-85% by weight, wherein the oil alone has a viscosity of above 305 Pa.s at 20°C.

9. (Once Amended) The gasification process for preparing synthesis gas, wherein a liquid bituminous oil is obtained according to the process of claim 1 and wherein said liquid bituminous oil, having a temperature of above 140°C, i s fed to a gasification unit in which synthesis gas is obtained.

10. (Once Amended) The use of the oil phase obtained in the process of claim1 having a temperature of above 140 °C as feedstock in a gasification process.

Respectfully submitted,
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MARKED UP VERSION OF AMENDED CLAIMS

2. (Amended) The [P] process [according to] of claim 1, wherein in step (a) the temperature is raised to a value of between 140-200°C.

3. (Amended) The [P] process [according to] of claim 2, wherein in step (a) the temperature is raised to a value of between 160-200°C.

4. (Amended) The [P] process [according to] of claim 3, wherein the temperature of the resulting mixture is raised from a value of between 120-150°C t o a value of between 160-180°C by making use of the indirect heat exchange means.

5. (Amended) The [P] process [according to any one] of claim[s] 1[-4], wherein the pressure in step (b) is sufficiently high in order to obtain both phases in the liquid state.

6. (Amended) The [P] process [according to] of claim 5, wherein in step (b) the liquid water phase has a pH of below 7.

7. (Amended) The [P] process [according to] of claim 6, wherein the pH of the liquid water phase is between 4 and 6.

8. (Amended) The [P] process [according to any one] of claim[s] 1[-7], wherein the starting emulsion has a water content of between 1-40% by weight, a surfactants content of between 0.01-5% by weight and an oil content of between 60-85% by weight, wherein the oil alone has a viscosity of above 305 Pa.s at 20°C.

9. (Amended) The [G] gasification process for preparing synthesis gas, wherein a liquid bituminous oil is obtained according to the process [according to any one] of claim[s] 1[-8] and wherein said liquid bituminous oil, having a temperature of above 140°C, is fed to a gasification unit in which synthesis gas is obtained.

10. (Amended) The [U] use of the oil phase obtained in the process [according to any one] of claim[s] 1[-9] having a temperature of above 140 °C as feedstock in a gasification process.

BREAKING OF OIL/WATER EMULSION

The invention is related to a process for breaking an emulsion of bituminous oil and water into the respective bituminous oil and water phases.

In the Orinoco Basin, in Trinidad, in North America, and in other areas, deposits of heavy oil and asphalt occur which are noted for their high bitumen content. These natural substances -which resemble oil and are commonly known as bituminous oil- can only be extracted by processes reducing viscosity and not by standard refinery methods. The extraction method currently used in the Orinoco Basin comprises emulsification of the bituminous oil at bed level, extraction of the emulsion, upgrading and transport.

Emulsifying the bituminous oils with water brings about a considerable reduction in the viscosity of these oils. The original viscosity in excess of 300 Pa.s at 20 °C is reduced to the range of 12 to 35 Pa.s as a consequence of emulsification. It is only this viscosity-reducing process that permits the extraction, transport and further processing of bituminous oil. Owing to the relatively high bitumen content, the bituminous oil cannot be easily processed by traditional refinery methods.

At present, the bituminous oil emulsion is used to fire power stations. The high sulphur content in bituminous oils (from 3 to 4%) causes a correspondingly high level of environmental pollution, a level which is becoming more and more unacceptable in the industrialised countries. The alternative is to produce desulphurized fuel gas by partial oxidation, also referred to as gasification, of the bituminous oil, thus obtaining raw

gas mainly consisting of CO and H_2. The raw gas is subsequently treated to obtain desulphurized fuel gas suitable for firing combined cycle power plants. The partial oxidation of bituminous oil is also suitable for the generation of synthesis gas or hydrogen, which can be used in a Fischer-Tropsch process or in processes for preparing a wide range of chemicals such as methanol, ammonia, oxy-products, formic acid and acetic acid.

EP-A-790292 describes a process in which an emulsion of Olinoco tar and water, also containing small amounts of sulfonic acid type surface active agent, having a starting temperature of 20 to 30 °C is broken by raising the temperature of the emulsion to 150 °C in two stages by means of an indirect heat exchange.

US-A-5441548 also describes a process in which a bituminous oil/water emulsion is broken by raising the temperature of the emulsion to a temperature of between 130 and 170 °C by means of two heat exchangers in series. The water and bituminous oil phase are subsequently separated by means of phase separation in a gravity type emulsion separator. According to the specification no additional chemicals such as for example demulsifiers are added to improve the separation of the bituminous oil phase and the water phase.

A disadvantage of the above processes is that the starting emulsion still has a relatively high viscosity at a temperature of below 100 °C when entering the first heat exchanger. Because of this high viscosity the heat exchanger must be equipped with large diameter tubes through which the emulsion flows and/or a high pressure must be applied to overcome the pressure drop in the first part of the heat exchanger where the viscosity is still high. Large diameter tubes are less effective resulting in that the heat exchanger will become large in

order to perform the desired raise in temperature or, as is the case in US-A-5441548, more than one heat exchanger will have to be used in series. The high pressure needed is disadvantageous because special pumps must be used. Furthermore the heat exchanger and the process equipment downstream of the heat exchanger, like for example the gravity-type emulsion separator, must be designed for this higher pressure level for obvious safety reasons. The present invention provides a process, which can be operated at a lower pressure having all the obvious advantages in view of the above.

The following process achieves this object. Process for separating an emulsion of a bituminous oil and water into a liquid water phase and a liquid bituminous oil phase, wherein the following steps are performed:

(a) raising the temperature of the bituminous oil/water emulsion having a temperature of below 100 °C to a temperature of above 140 °C, and

(b) performing a phase separation wherein a liquid water phase and a liquid bituminous oil phase are obtained,

wherein the heating of the emulsion in step (a) is effected by first mixing part of liquid bituminous oil phase obtained in step (b) having a temperature of above 140 °C with the bituminous oil/water emulsion and subsequently raising the temperature of the resulting mixture to a temperature of above 140 °C by making use of indirect heat exchange means.

It has been found that by mixing the emulsion feed with part of the bituminous oil phase obtained in the phase separation of the emulsion the temperature can be sufficiently raised in order to lower the viscosity of

the mixture entering the heat exchanger means. This results in that a lower pressure drop in the heat exchanger has to be overcome enabling a lower inlet

pressure. Accordingly smaller and more simple pumps, smaller heat exchanger means and process equipment designed for lower pressure levels can be used with the process according the invention.

Applicants have also found that the temperature at which step (b) is performed is important for an efficient process. Applicants found that for this oil-water system the water phase has a higher density than the oil phase at temperatures of below about 130 °C. Above about 130 °C the oil phase has a higher density than the water phase. By increasing the temperature starting from about 130 °C the difference in density increases and thus the ease at which the phases separate in a heavy oil phase and a lighter water phase increases. At 140 °C a sufficient difference is achieved to perform a phase separation. Preferably the temperature is not higher than 200 °C because at higher temperatures the solvability of the oil in water and water in oil becomes undesirably high. A more preferred range is between 160-200 °C in which the difference in density is sufficiently high to achieve a efficient phase separation and the solvability is of water in oil and oil in water is within an acceptable range. Most preferably the temperature is between 160-180 °C. The weight ratio of oil phase and emulsion which are mixed to achieve the first raise in temperature is preferably between 1 to 2 and 1 to 5.

Suitable indirect heat exchanger means to be used in the process according to the invention can be for example those means disclosed in US-A-5441548. An example of a preferred heat exchanger means is a shell-tube heat exchanger, wherein a hot medium, like for example steam or hot oil, at the shell side exchanges its heat with the mixture comprising the emulsion present at the tube side. The temperature rise in the heat exchanger means is

preferably from between 120-150 °C to a value between 160-180 °C.

For some applications of the bituminous oil obtained by the process according to the invention it is advantageous to lower the level of water soluble salts in said oil. Examples of such salts are magnesium, calcium, sodium, potassium, containing salts. Such salts may cause severe fouling in for example the process equipment of a partial oxidation process. In for example the process disclosed in US-A-5441548 the content of such water soluble salts in the bituminous oil phase will be too high for these applications. Applicants have now found that by lowering the pH of the water phase obtained in step (b) to a level of below 7 a lower content of these salts remain in the oil phase. The pH is preferably between 4 and 6. Preferably the pressure during phase separation is sufficiently high to ensure that the water phase is obtained as a liquid in step (b). Suitable pressures are between 5 and 20 bars. Obtaining the water phase as a liquid further ensures that most salts will be removed with the water phase. The improved removal of these water soluble salts by lowering the pH of the water phase is preferably used in combination with the process according to the invention. It must be appreciated that this technical measure can also be used in a more general manner and not only limited as an embodiment of the present invention in which the recycling of part of the oil is an essential feature. Typical emulsions which can be treated by the above preferred embodiment may have a calcium content of above 20 ppmw and/or a magnesium content of above 20 ppmw.

Typically the bituminous oil/water emulsion has a water phase with a pH of above 7 caused by the natural and/or the added surfactants present in the emulsion. In order to achieve the lower pH suitably an acid is added

prior to phase separation. Examples of suitable acids which may be used are those acids which do not cause significant problems in the water treatment facilities in which the water phase is suitably further treated before being returned to surface water, like rivers, sea or lakes. Examples of such acids are sulphuric acid, phosphoric acid and acetic acid of which sulphuric acid is preferred because of its availability and ability to simply be removed from the water phase, for example as gypsum. The amount of acid added can be easily determined by measuring the pH of the water phase obtained in the process.

Phase separation can be performed in any conventional phase separation device known to one skilled in the art. These devices can be a gravity-type separator or a combination of a gravity separator and a down stream emulsion separator in the electrostatic field.

The bituminous oil can be the natural occurring crude petroleum sources found in the Orinoco Basin, in Trinidad, in North America, and in other areas, which are noted for their high bitumen content. The bituminous oil may also be the vacuum residue fraction obtained when refining a typical petroleum crude. Due to for example local environmental reasons it may happen that these fractions cannot be further processed to fuels. It may be desirable to transport these fractions to locations where they may be used as gasification feedstock. Due to their viscous properties these fractions will be suitably transported as a water/oil emulsion.

The bituminous oil/water emulsion may also comprise surfactants. Examples of possible surfactants are ethoxylated alkyl phenols, for example nonyl phenol ethoxylate compounds, ethoxylated alcohols, water soluble amine compounds, alkali compounds and combinations thereof. Examples of water soluble amines are ethylamine,

diethylamine, triethylamine, n-butylamine, tri-isobutylamine, dimethylamine, methylamine, propylamine, dipropylamine, sec-propylamine, butylamine, sec-butylamine, ethanolamine and mixtures thereof. The ethoxylated alcohols may contains between 12-18 carbon atoms, for example polyethoxylated tridecanol. Examples of alkali compounds are sodium chloride, potassium chloride, sodium nitrate, potassium nitrate, calcium nitrate, magnesium nitrate and mixtures thereof. The emulsion typically comprises between 60-85% by weight of the bituminous oil, between 0.01 to 5% by weight of surfactant and between 10 to 40% by weight of water. Examples of bituminous oil water emulsions optionally comprising the exemplified surfactants, which can be used in the process according to the present invention are for example described in US-A-5419852, US-A-5437693, US-A-5480583, US-A-5503772, US-A-5556574, US-A-5603864 and US-A-5622920. Preferred emulsion are ORIMULSION and OLIMULSION as for example described in US-A-4795478 or EP-A-790292 respectively (ORIMULSION is a trademark of Intevep S.A. Venezuela) (OLIMULSION is a trademark of Bitumes Olinoco S.A. Venezuela).

The bituminous oil obtained in the above process can be advantageously be used as feedstock for a gasification process. The gasification process can be any process known in the art which is suitable for processing heavy feedstocks like the above described bituminous oil. Examples of such processes are the SHELL Gasification Process as for example described by Heurich et al. in "Partial Oxidation in the Refinery Hydrogen Management Scheme", AIChE 1993 Spring Meeting, Houston, 30 March 1993 and the TEXACO process as described in Petroleum Review June 1990, page 311-314. Typically the bituminous oil and oxygen or air is fed to a gasification burner. Optionally a moderator gas, for example steam or

carbon dioxide, may be supplied to the burner as well. At the burner outlet the reactants are atomised and mixed and an exothermic partial oxidation takes place at a temperature of between 1300 and 1500 °C. The pressure is typically between 10 and 90 bar. The resulting fuel or synthesis gases will mainly consist of CO and H_2. Other components will be CO_2, CH_4, H_2O, H_2S, COS, N_2 and Ar. The hot fuel gases are subsequently lowered in temperature for example by means of a water quench or by means of indirect heat exchange. An example of such an indirect heat exchange method is described in EP-A-774103 and which describes a vertical oriented shell-tube heat exchanger in which the temperature is typically lowered from 1300-1500 °C to a temperature typically between 300 and 350 °C. In such a device high pressure steam is generated at the shell side of the unit while the synthesis gases are lowered in temperature at the tube side of the heat exchanger. The advantages of the invention are even more achieved when the bituminous oil obtained by the present invention is used in a gasification process which applies such an indirect heat exchanger. This is because the water soluble salt in the feedstock, which salts are effectively removed by one preferred embodiment of the present invention, can cause severe plugging of the heat exchanger (tubes).

The invention will be illustrated by making use of Figure 1. Figure 1 describes a process for separating a bituminous oil/water emulsion and the gasification of the bituminous oil thus obtained. A bituminous oil/water emulsion (1) is mixed with a recycle stream of bituminous oil (8) resulting in stream (2). This mixture (2) is heated to the desired temperature in heat exchanger (3) resulting in heated stream (4) which is fed to the gravity phase separator (5). In gravity-type

separator (5) a water phase (6) as the top phase and a heavier bituminous oil phase (7) as the bottom phase is obtained. Part of the bituminous phase (7) is recycled via pump (9) to heat exchanger (3) as stream (8) to which recycle stream some acid is supplied via stream (10) in order to lower the pH to the desired level. The remaining part (11) of the bituminous oil phase (7) is fed to gasification unit (14). To this reactor unit comprising burners (not shown) also air or oxygen is fed via (12) and optionally a moderator gas is fed via (13). The fuel gas mixture obtained via (15) is lowered in temperature in a shell-tube heat exchanger (16) to which boiler feed water is supplied to via (17) and high pressure steam (18) is generated. Fuel gas mixture (19) obtained in heat exchanger (16) is further processed in downstream unit operations (not shown) in which for example ash and soot and sulphur containing compounds are removed.

The invention will be illustrated by the following non-limiting example.

Example 1

785 ton/day of ORIMULSION having a temperature of 40 °C was contacted with 3140 ton/day of recirculating oil phase having a temperature of 170 °C at 15 bar resulting in a mixture having a temperature of 149.5 °C. This mixture was further heated in a heat exchanger to a temperature of 170 °C having a viscosity of 179 cSt. Phase separation was performed resulting in 236 ton/day water and 3690 ton/day oil phase at 10 bar of which oil phase 3140 ton/day is used to heat the Orimulsion feed and 550 ton/day is obtained as oil product having a temperature of 170 °C and a viscosity of 102 cSt. The pressure of the circulating oil phase is raised from 10 bar to 15 bar by means of a pump. To this circulating mixture 63 kg/day of sulphuric acid is added.

Example 2

To one weight part of a typical ORIMULSION (ORIMULSION is a trade name of Intevep S.A. describing an emulsion of a bituminous oil and water and their preparation is described in US-A-4795478) an amount of sulphuric acid was added in order that the pH of the resulting water phase had a pH of 5 (80 mg sulphuric acid per kg of emulsion). The water content of the emulsion was 30% by weight. Phase separation was performed at a temperature of 180 °C and at a pressure of 10 bar. The phases remained in the liquid phase during phase separation. A water phase was obtained as the top phase. In Table 1 some more information of the ORIMULSION used and the resulting phase separation is presented.

Example 3

Example 2 was repeated except that no acid was added. The pH of the resulting water phase was 7.9. See also Table 1.

Table 1

	Orimulsion	Example 2 Bituminous oil phase	Example 3 Bituminous oil phase
Calcium (ppmw)	34	12	52
Magnesium (ppmw)	46	5	46
oil in water phase after phase separation (wt%)	-	0.1	0.3

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AMENDED SHEET

TS 0854 PCT

NEW CLAIMS

1. Process for separating an emulsion of a bituminous oil and water into a liquid water phase and a liquid bituminous oil phase, wherein the following steps are performed:

(a) raising the temperature of the bituminous oil/water emulsion having a temperature of below 100 °C to a temperature of above 140 °C, and

(b) performing a phase separation wherein a liquid water phase and a liquid bituminous oil phase are obtained, wherein the heating of the emulsion in step (a) is effected by first mixing part of liquid bituminous oil phase obtained in step (b) having a temperature of above 140 °C with the bituminous oil/water emulsion and subsequently raising the temperature of the resulting mixture to a temperature of above 140 °C by making use of indirect heat exchange means.

2. Process according to claim 1, wherein in step (a) the temperature is raised to a value of between 140-200 °C.

3. Process according to claim 2, wherein in step (a) the temperature is raised to a value of between 160-200 °C.

4. Process according to claim 3, wherein the temperature of the resulting mixture is raised from a value of between 120-150 °C to a value of between 160-180 °C by making use of the indirect heat exchange means.

5. Process according to any one of claims 1-4, wherein the pressure in step (b) is sufficiently high in order to obtain both phases in the liquid state.

6. Process according to claim 5, wherein in step (b) the liquid water phase has a pH of below 7.

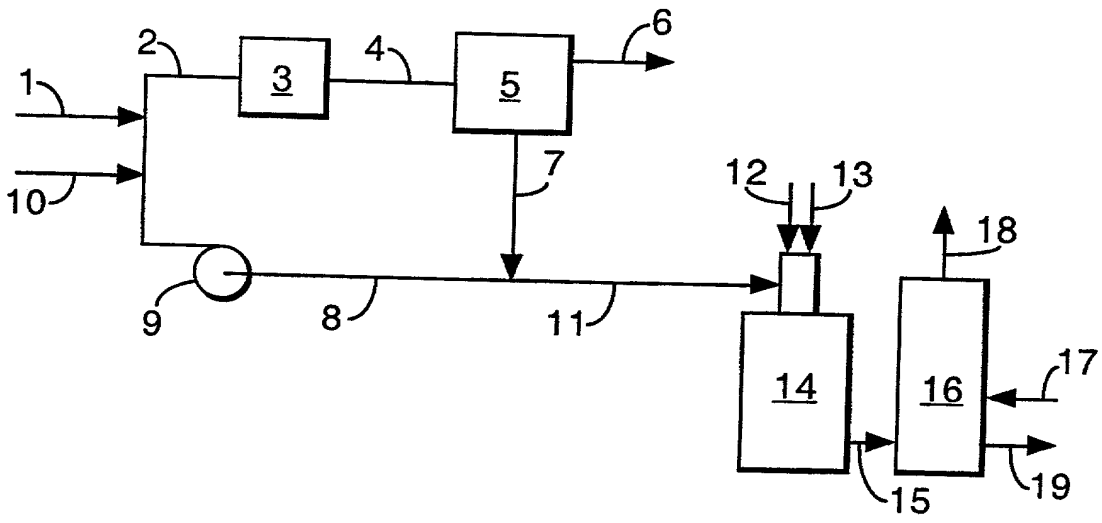
AMENDED SHEET

7. Process according to claim 6, wherein the pH of the liquid water phase is between 4 and 6.

8. Process according to any one of claims claim 1-7, wherein the starting emulsion has a water content of between 10-40% by weight, a surfactants content of between 0.01-5% by weight and an oil content of between 60-85% by weight, wherein the oil alone has a viscosity of above 305 Pa.s at 20 °C.

9. Gasification process for preparing synthesis gas, wherein a liquid bituminous oil is obtained according to the process according to any one of claims 1-8 and wherein said liquid bituminous oil, having a temperature of above 140 °C, is fed to a gasification unit in which synthesis gas is obtained.

Fig.1.



DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

ATTORNEY'S DOCKET NO.
TS 0854 USA P

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FULL NAME OF SOLE OR FIRST INVENTOR (given name, family name)
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INVENTOR'S SIGNATURE — DATE SIGNED
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FULL NAME OF FIFTH JOINT INVENTOR, IF ANY (given name, family name)

FIFTH INVENTOR'S SIGNATURE — DATE SIGNED

RESIDENCE — CITIZENSHIP

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TS 0854 USA P

DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name. I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled Breaking oil / water emulsion the specification of which is attached hereto unless the following box is checked:

[XX] was filed on 25 July 2000 as United States Application Number or PCT International Application Number PCT/EP00/07174 and was amended on 12 July 2001 (if applicable).

I hereby state that I have reviewed and understand the contents of the above-identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. § 119(a)-(d) or § 365(b) of any foreign application(s) for patent or inventor's certificate, or § 365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below by checking the box, any foreign application for patent or inventor's certificate, or PCT International application having a filing date before that of the application on which priority is claimed.

PRIOR FOREIGN APPLICATION(S)

APPLICATION NUMBER	COUNTRY	DAY/MONTH/YEAR FILED	Priority Not Claimed
99305908.8	**EP**	**26 July 1999**	[ ]
APPLICATION NUMBER	COUNTRY	DAY/MONTH/YEAR FILED	[ ]

I hereby claim the benefit under 35 U.S.C. § 119(e) of any United States provisional application(s) listed below.

APPLICATION SERIAL NO.	FILING DATE
APPLICATION SERIAL NO.	FILING DATE

I hereby claim the benefit under 35 U.S.C. § 120 of any United States application(s) or § 365(c) of any PCT International application designating the United States, listed below and insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT International application in the manner provided by the first paragraph of 35 U.S.C. § 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR § 1.56 which became available between the filing date of the prior application and the national or PCT International filing date of this application.

APPLICATION SERIAL NO.	FILING DATE	STATUS-PATENTED, PENDING, ABANDONED
APPLICATION SERIAL NO.	FILING DATE	STATUS-PATENTED, PENDING, ABANDONED

I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

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